
1-(4-सल्फोफिनाइल)-3-कार्बोक्सी-5-
पाइराजोलोन — विशिष्टि
(पहला पुनरीक्षण)

1-(4-Sulphophenyl)-3-Carboxy-5-
Pyrazolone — Specification
(First Revision)

ICS 71.080.99

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
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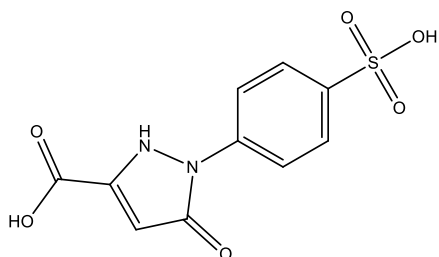
August 2023

Price Group 6

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

1-(4-sulphophenyl)-3-carboxy-5-pyrazolone ($C_{10}H_8N_2O_6S$), is an important dye intermediate used in the manufacture azo dyes. It has the following structural formula:



1-(4-SULPHOPHENYL)-3-CARBOXY-5-PYRAZOLONE
(MOLECULAR MASS: 284)
CAS NUMBER:118-47-8

This standard was first published in 1977. In this revision, determination of assay by nitrite value and HPLC have been incorporated. Further, limit of requirement of matter insoluble in sodium hydroxide solution has been modified.

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as given in Annex C, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

1-(4-SULPHOPHENYL)-3-CARBOXY-5-PYRAZOLONE — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements, the methods of sampling and test for 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone.

2 REFERENCES

The standards given below contain provisions which through reference in the text constitute provisions of this standard, at the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)
IS 14887 : 2014	Textiles — High density polyethylene (HDPE)/polypropylene (PP) woven sacks for packaging of 50 kg food grains — Specification (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of beige powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements as given in Table 1, when tested according to the methods prescribed col (4) and col (5) of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in HDPE/PP woven sacks (*see* IS 14887). Each bag shall be securely closed.

4.2 Marking

4.2.1 Each bag shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer/supplier, complete address and his recognized trade-mark, if any;
- c) Gross, net and tare mass;
- d) Batch number, month and year of manufacturing;
- e) GHS label (pictogram, signal word, hazard statement and precautionary statement);
- f) Shelf life of the material; and
- g) Any other statutory requirement.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for purity by nitrite value shall be conducted on each of the individual samples.

5.2.2 Tests for determination of all other characteristics, namely assay by HPLC and matter insoluble in sodium hydroxide solution, given under Table 1, shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples

The lot shall be declared as conforming to the requirement of assay by nitrite value if each of the individual test results satisfies the relevant requirements given in Table 1.

Table 1 Requirement for 1-(4-Sulphophenyl)-3-Carboxy-5-Pyrazolone

(Clauses 3.2, 5.2.2, 5.3.1, 5.3.2 and 6.2)

SI No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Cl No. of IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Assay (by nitrite value), percent by mass (on dry basis), <i>Min</i> or	75.0	A	-
ii)	Assay (by HPLC ¹⁾), percent area (on dry basis), <i>Min</i>	99.0	B	-
iii)	Matter Insoluble in sodium hydroxide solution, percent by mass, <i>Max</i>	0.30	-	11.2

¹⁾In case of disputes, determination of assay by HPLC, shall be the referee method.**5.3.2 For Composite Samples**

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements in Table 1.

6 TEST METHODS**6.1 Preparation of sample**

Dry the material at $90\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ to constant mass. Grind and mix well. Transfer the material to a wide

mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

6.2 Test shall be carried out according to the methods prescribed in col (4) and (5) of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (i)]

**DETERMINATION OF 1-(4-SULPHOPHENYL)-3-CARBOXY-5-PYRAZOLONE CONTENT (ASSAY)
BY NITRITE VALUE**

A-1 REAGENTS**A-1.1 Concentrated Hydrochloric Acid****A-1.2 Potassium Bromide****A-1.3 Standard Sodium Nitrite Solution — 0.1 N****A-1.4 Potassium Starch Iodide Indicator Paper****A-1.5 Ice****A-2 PROCEDURE**

Weight 10 g to 14 g dry powder in 250 ml glass beaker. Add distilled water approximately 150 ml and stir with glass rod to make a smooth slurry. Add 20 percent soda ash solution (approximately 7 ml to 10 ml) to dissolve the powder to make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 ml of the solution by using pipette in to 1 000 ml beaker.

Add 200 ml to 250 ml distilled water. Add ice cubes to make the temperature around 10 °C. Weigh and add 1 g potassium bromide into the cold solution. Add hydrochloric acid to make the pH acidic (pH around 2 to 2.5 on pH paper) approximately 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the endpoint to put the spot-on starch iodide paper, the end point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

A-3 CALCULATION

Assay (by nitrite value), percent by mass (on dry basis) = $\frac{V \times N \times 284}{M}$

where

V = volume, in ml, of standard sodium nitrite solution used in the titration;

N = normality of sodium nitrite solution; and

M = mass, in g, of the dry material taken for the test.

ANNEX B

[Table 1, *Sl No.* (ii)]

**DETERMINATION OF 1-(4-SULPHOPHENYL)-3-CARBOXY-5-PYRAZOLONE CONTENT (ASSAY)
BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)**

B-1 OUTLINE OF METHOD

High performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 HPLC — isocratic liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.1.1 Column — C18 column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-2.2 Analytical balance**B-3 REAGENTS**

B-3.1 Tetrabutylammonium hydrogen sulphate — HPLC grade

B-3.2 Dipotassium hydrogen phosphate — analytical grade (AR)

B-3.3 Acetonitrile — HPLC grade

B-3.4 Water — HPLC grade

B-3.5 1-(4-Sulphophenyl)-3-Carboxy-5-Pyrazolone — of known purity

B-4 SAMPLE PREPARATION

Weigh accurately 0.010g (10 mg) of dry 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone in 100 ml volumetric flask. Dissolve it in water and

make up to 100 ml with water.

B-5 BUFFER PREPARATION

Take 12 g tetrabutylammonium hydrogen sulphate (TBAHS) and 29 g dipotassium hydrogen phosphate (K₂HPO₄) in 190 ml water and dissolve (stock solution).

B-6 FLOW RATE — 1 ml/min

B-7 MOBILE PHASE — 100 ml water + 4 ml buffer + 40 ml acetonitrile

B-8 COLUMN OVEN TEMPERATURE — 26 °C

B-9 INJECTION VOLUME — 20 µl

B-10 RUN TIME — 12 min, *Max*

B-11 WAVELENGTH — 254 nm

B-12 PEAK TIME — 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone: 5.5 min

B-13 CALCULATION

Calculate the peak area of individual constituent pertaining to 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone from the chromatogram of the material. the concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone.

$$1-(4\text{-sulphophenyl})\text{-}3\text{-carboxy-}5\text{-pyrazolone} = \frac{A}{\text{Total Area}} \times 100$$

where

A = area of peak 1-(4-sulphophenyl)-3-carboxy-5-pyrazolone in sample.

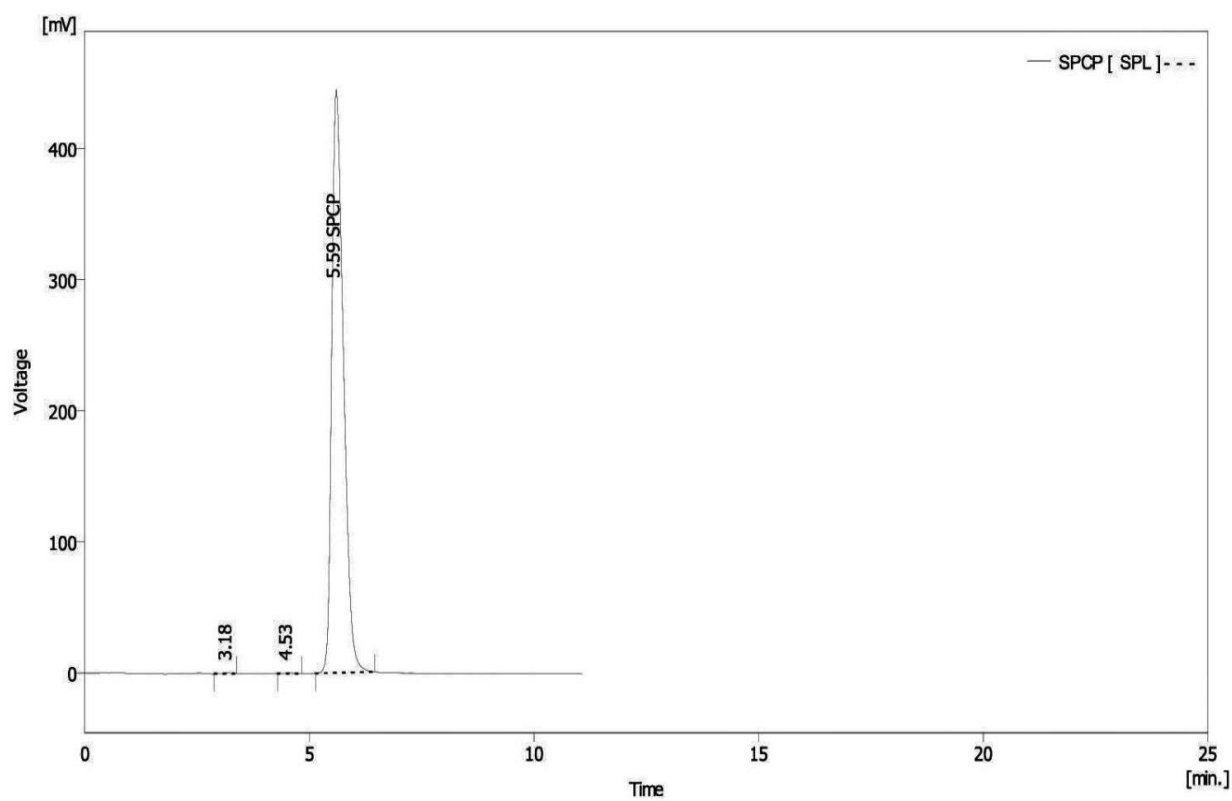


FIG. 1 TYPICAL CHROMATOGRAM

ANNEX C

(Foreword)

PICTOGRAMS, SIGNAL WORD, HAZARD STATEMENT AND PRECAUTIONARY STATEMENT

Pictogram(s)	: Nil
Signal word	: Nil
Hazard statement(s)	: Nil
Precautionary statement(s)	General: P101: If medical advice is needed have product container or label at hand. P102: Keep out of reach of children. P103: Read label before use. Prevention: P280: Wear protective gloves/protective clothing/eye protection/face protection. Response: P310: Immediately call a poison center/doctor.

ANNEX D*(Foreword)***COMMITTEE COMPOSITION**

Dye Intermediates Sectional Committee, PCD 26

<i>Organization</i>	<i>Representative(s)</i>
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING (Chairperson)
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Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI KIRAT PATEL (<i>Alternate</i>)
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Deepak Nitrite Limited, Vadodara	SHRI SAILASH RAVAL SHRI RAJENDRA SHINDE (<i>Alternate</i>)
Defence Research Development Organization, Ministry of Defence, New Delhi	DR PRADEEP K. GUPTA
Dystar, Mumbai	DR MONIKA SINGH
Ecological and Toxicological Association of Dyes, Vadodara	DR PARITI SIVA RAMA KUMAR
Gujarat Dyestuffs Manufacturers Association, Ahmedabad	SHRI YOGESH PARIKH SHRI ANIL M. JAIN (<i>Alternate I</i>) SHRI HARESH BHUTA (<i>Alternate II</i>)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	SHRI R. M. PATEL SHRI C. S. PATEL (<i>Alternate</i>)
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Huntsman Textile Effects, Mumbai	MS PRACHI JOSHI
Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN

<i>Organization</i>	<i>Representative(s)</i>
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The Dyestuff Manufactures Association of India Office, Mumbai	SHRI V. R. KANETKAR
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Member Secretary
SHRI ABHIJEET SINGH
SCIENTIST 'B'/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : 601/A, Konnectus Tower -1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	{ 2323 7617
Eastern : 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	{ 2367 0012 2320 9474
Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	{ 265 9930
Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	{ 2254 1442 2254 1216
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